Lehigh University
Emulsion Polymers Inst

Quarterly Progress Report - May 20-August 19, 1987

"Extension of NAS8-36286: "Evaluation of Rotating-Cylinder and Piston-Cylinder Reactors for Ground-Based Emulsion Polymerization"

to

National Aeronautics and Space Administration
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama 35812

by

J. W. Vanderhoff and M. S. El-Aasser
Emulsion Polymers Institute
Lehigh University
Bethlehem, Pennsylvania 18015

Objectives

The objectives of this program are to apply ground-based emulsion polymerization reactor technology developed under NASA sponsorship to improve the production of: 1. monodisperse latex particles for calibration standards, chromatographic separation column packing, and medical research; 2. commercial latexes such as those used for coatings, foams, and adhesives.

Introduction and Background

This project comprises a one-year collaborative effort of the Emulsion Polymers Institute of Lehigh University and Marshall Space Flight Center to design, fabricate, test, and operate two new polymerization reactor designs and assess their potential for commercial development.

Today, monodisperse latexes of 0.1-2 micrometer size are sold in the United States by at least four companies. These latexes are made by seeded emulsion polymerization, with the product of one polymerization used as the seed for the next. This technique gives increasing amounts of coagulum with increasing particle size greater than 2 micrometers, so that it is difficult to prepare these larger sizes. NASA-MSFC has sponsored the Lehigh program to produce large-particle-size monodisperse latexes in space. The rationale for the preparation in space was that the monomer-swollen particles would neither cream nor settle in microgravity and thus the agitation need be sufficient only to provide good heat transfer and mixing, resulting in decreased mechanical shear and hence reduced coagulum. This rationale was demonstrated unequivocally on Shuttle flights STS-3, STS-4, STS-6, STS-7, and STS-11: particles of 5-30 micrometers diameter and 1.0-1.4% coefficients of variation were produced with negligible coagulum, as compared with the 2-5% coefficients of variation of the corresponding ground-based latexes, which invariably contained large amounts of coagulum. The National Bureau of Stardards has accepted the 10 micrometer STS-6 and the two 30 micrometer STS-11 latexes as Standard Reference Materials, to make them the first products made in space for sale on earth. This MSFC-Lehigh program has also been

(NASA-CR-179179) EVALUATION OF ROTATING-CYLINDER AND PISTON-CYLINDER REACTORS FOR GROUND-BASED EMULSION POLYMERIZATION Quarterly Progress Report, 20 May - 19 Aug. 1987 (Lehigh Univ.) 11 p
N87-29668
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successful in terms of: 1. the development, construction, and operation of the MLR piston-cylinder reactors; 2. the development of laboratory processing techniques; 3. the development and improvement of the ground-based polymerization recipes to produce particles up to 18 micrometers diameter with high monodispersity; 4. the demonstration of a market for these products.

The ground-based production of these large-particle-size monodisperse latexes is of interest to prepare quantities sufficient for uses less demanding than calibration, e.g., chromatographic separation column packing; however, the formation of coagulum remains a serious problem. In the early stages of polymerization, the soft, sticky monomer-swollen particles tend to cream, and increasing the agitation rate to offset this creaming causes more violent particle-particle collisions, resulting in flocculation and coagulation of the particles. A more-gentle-but-thorough method of agitation must be developed to produce these large-particle-size latexes on earth.

The space and ground-based polymerizations in the MLR 100-ml piston-cylinder reactors showed that the formation of coagulum in monodisperse latexes was alleviated by the slow stirring rate and uniform mixing of the H-blade stirrer. Thus this reactor design can serve as the basis for scaleup of ground-based emulsion polymerizations, not only of the large-particle-size monodisperse latexes, but also of commercial latexes of the types used for latex paints, paper coatings, carpet backing, nonwoven fabrics, and pressure-sensitive adhesives. For many years, these commercial latexes have been produced in stirred-tank reactors with varying levels of coagulum, and few new reactor designs have been tried. Throughout this time, the formation of coagulum has remained a serious problem. Therefore, any new reactor design such as the piston-cylinder reactor which promises to alleviate the formation of coagulum should be evaluated.

Another new reactor design which promises to alleviate the formation of coagulum is the rotating-cylinder reactor. Several years ago, D. M. Kornfeld (MSFC) proposed that ground-based emulsion polymerizations could be carried out using a horizontally-mounted rotating-cylinder reactor to simulate microgravity. This reactor design gives less mechanical shear during polymerization than the stirred-tank reactor. MSFC built a 235-ml prototype rotating-cylinder reactor and used it to conduct polymerizations. Lehigh University used this reactor to prepare monodisperse polystyrene latexes with diameters as large as 62 micrometers, one of the largest particle sizes prepared to date. The uniformity of these latexes was poorer than that of those made in space, possibly because of lower-quality seed latex and temperature gradients within the reactor; however, it is probable that the uniformity can be improved considerably. Commercial latexes of the types used for paints, paper coatings, carpet backing, nonwoven fabrics, and pressure-sensitive adhesives could also be produced in a rotating-cylinder reactor with minimal mechanical shear and hence less coagulum. The latter applications require consideration for scaleup of the rotating-cylinder and piston-cylinder reactors to produce commercial volumes.

Therefore, NASA sponsored the present program to design and construct three new two-liter polymerization reactors, two rotating-cylinder reactors scaled up from the MSFC 235-ml reactor and one pis-

ton-cylinder reactor scaled up from the MLR reactors, and to demonstrate the applicability of these reactors to produce on earth: 1. large-particle-size monodisperse latexes; 2. commercial latexes of the types used for coatings.

The Quarterly Progress Report (February 20, 1986-May 20, 1987) described: 1. the Second Design Review held January 24, 1987; 2. the Third MCS, Inc. Monthly Progress Report, February 28, 1987; 3. the Third Design Review held March 21, 1987; 4. the Fourth MSC, Inc. Monthly Progress Report, March 31, 1987; 5. the Fourth Design Review including a discussion of the operational plan for selection of the vendors and construction of the reactor component parts held April 6, 1987; 6. the Fifth MSC, Inc. Monthly Progress Report, April 30, 1987; 7. the First Fabrication Review including a discussion of the detailed purchasing plan held May 11, 1987.

Progress

During this quarter, Lehigh and MSC, Inc. further developed and revised the design of the reactors, developed plans for the procurement of the mechanical and electronic reactor parts, selected the vendors to supply these parts, ordered the parts from the selected vendors, and received most of the completed parts.

The First Fabrication Review attended by F. A. Vicente, M. S. El-Aasser, and D. H. Nyby was held on May 11 to discuss the details of all of the drawings and to develop a detailed purchasing plan to optimize the expenditure of the project funds. The drawings were clarified and interpreted, and the parts lists were reviewed to facilitate the ordering of parts to the drawings. It was decided to delay the fabrication of the electronic components until all of the requisite parts have been received. This meeting was summarized in the Sixth MSC, Inc. Monthly Progress Report of May 31 (Appendix A).

As a result, the drawings of the mechanical parts were forwarded to the vendors with a request for bids. Once all of the bids were received, the vendors were selected and purchase orders were placed. The delivery dates for the mechanical parts were set for June 12, 1987. Also, a preliminary list of the electronic parts was forwarded to the vendors with a request for bids. The final list of electronic parts was received from MSC, Inc. on June 9, 1987. This list was also forwarded to the vendors with a request for bids. Once all of the bids were received, the vendors were selected and purchase orders were placed.

The Second Fabrication Review attended by F. A. Vicente, C. G. Hunt, M. S. El-Aasser, D. H. Nyby, and J. W. Vanderhoff was held on June 29 to examine the mechanical parts received and coordinate the procurement of the remaining requisite mechanical and electronic parts. By this date, about 60% of the requisite mechanical parts had been received. The list of the remaining requisite parts was reviewed. It was decided to delay the fabrication of the electronic components until all of the requisite parts have been received. This meeting was summarized in the Seventh MSC, Inc. Monthly Progress Report of June 31 (Appendix B).

The Third Fabrication Review attended by F. A. Vicente, M. S.

El-Aasser, E. D. Sudol, D. H. Nyby, and J. W. Vanderhoff was held on July 11 to examine the mechanical parts received and coordinate the procurement of the remaining requisite mechanical and electronic parts. By this date, about 75% of the requisite mechanical parts had been received. The Fourth Fabrication Review attended by F. A. Vicente, E. D. Sudol, and D. H. Nyby was held on July 29 to examine the mechanical parts received and coordinate the procurement of the remaining requisite mechanical and electronic parts, and the Fifth Fabrication Review attended by F. A. Vicente, E. D. Sudol, and D. H. Nyby was held on July 30 to deal with the piston-cylinder problems described below. At the July 11 and 29 meetings, the list of the remaining requisite parts was reviewed. At the July 29 meeting, the electronic components and parts received were examined, and the procurement of the remaining requisite parts was reviewed. It was decided to delay the fabrication of the electronic components until all of the requisite parts have been received. These meetings were summarized in the Eighth MSC, Inc. Monthly Progress Report of July 31 (Appendix C).

At the July 11 and July 29 meetings, two problems became apparent: 1. the cylindrical insulation covers were thicker than specified in the drawings; 2. the pistons did not fit the cylinders.

The cylindrical insulation covers were 0.25 inches thick, twice the 0.125-inch thickness specified in the drawings. This increased thickness makes the part heavier than planned; however, it does not affect its performance except that a greater weight must be handled in the assembly and disassembly of the reactor and a heavier-duty drive motor may be required. Therefore, at the July 30 meeting, it was decided to delay the ordering of the drive motor until the reactor was assembled and the effect of the heavier cylindrical insulation cover was assessed more thoroughly.

Also, it was found that the piston shaft did not fit into the LVDT housing assembly. Therefore, at the July 30 meeting, it was decided to:

1. Measure both the housing cylinder and the piston shaft;
2. Machine the housing cylinder to provide a slip fit between the two parts;
3. Repeat these steps for all three sets of matching components, if necessary, and tag the matching components;
4. Measure the inside diameter of the machined housing cylinders and reallodine the machined part.

This machining has now been completed.

Originally, it was planned to apply a Teflon coating to the inside wall of the cylinders to make the piston O-rings slip more easily; however, at the July 11 meeting, E. D. Sudol reported that similar Teflon wall coatings were abraded during polymerization experiments and that the latexes produced were contaminated with Teflon particles. Therefore, at the July 30 meeting, it was decided to hone and polish the inside cylinder walls to a #16, or possibly a #18, finish instead of having the Teflon coating applied. The honing and polishing would improve the slip of the piston O-rings but not as much as would the Teflon coating; however, if polished surface does

not give the requisite slip, the Teflon coating will be applied. This honing and polishing has now been completed.

The Sixth Fabrication Review attended by F. A. Vicente, E. D. Sudol, and D. H. Nyby was held on August 19 to examine the electronic parts received and begin the assembly of the electronic components.

The first reactor, complete with the electronic components, is scheduled to be assembled on September 20.

Financial

Total Amount Contract NAS8-36286	$ 157368
Total Expenditures through August 19, 1987	$ 132789
Estimated Funds to Completion	$ 24579
Anticipated Overrun	$ none

APPENDIX A

M S C INC.

2171 DAVIS DRIVE
MEDIA, PA 19063

Lehigh University
Emulsion Polymers Institute
Francis MacDonald Sinclair
Memorial Laboratory #7
Bethlehem, Pa 18015

31 May 1987

JUN 8 1987

Attn. Dr. J.W. Vanderhoff
Director

Dear Dr. Vanderhoff,

This submission constitutes our sixth monthly progress report under Lehigh University's P.O. 025379, dated 18 December 1986. It covers the period from 30 April 1987 through 31 May 1987.

Technical
Support to LU was given in regard to equipment/ parts acquisition. Clarification and interpretation of drawings and parts lists was given to permit and facilitate ordering of parts to the drawings.

Programmatic
A meeting was held at Lehigh U. on 11 May 1987 to discuss construction and parts acquisition.

An invoice for the effort to date is attached.

We are in receipt of the requested change order which authorizes us to continue support. We recognize that part of the effort (Electronics Fabrication) requires ordering and receipt of parts by LU. We will hold beginning construction until all the required electronic parts are available.
If you have any questions please feel free to call (215) 565-2947.

Sincerely,

F.A. Vicente
President

APPENDIX B

JUL 7 1987

M S C INC.

2171 DAVIS DRIVE
MEDIA, PA 19063

30 June 1987

Lehigh University
Emulsion Polymers Institute
Francis MacDonald Sinclair
Memorial Laboratory #7
Bethlehem, Pa 18015

Attn. Dr. J.W. Vanderhoff
Director

Dear Dr. Vanderhoff,

This submission constitutes our seventh monthly progress report under Lehigh University's P.O. 025379, dated 18 December 1986. It covers the period from 31 May 1987 through 30 June 1987.

Technical
Support to LU was given in regard to equipment/ parts acquisition. Clarification and interpretation of drawings and parts lists was given to permit and facilitate ordering of parts to the drawings. In addition support was given in a preliminary inspection of approximately 60% of the mechanical parts recei.ed a/0 29 June.

Programmatic
A meeting was held at Lehigh U. on 29 June 1987 to discuss construction, parts acquisition and inspect parts. Some parts appeared not to be constructed per our drawings. A meeting is scheduled for 11 July 1987 to inspect all mechanical parts received by LU a/o that date and decide disposition.

An invoice for the effort to date is attached.

We continue to hold beginning construction of the electrical s/s until all the required electronic parts are available.
If you have any questions please feel free to call (?:5) 565-2947.

Sincerely,

F.A. Vicente
President

M S C Inc

2171 Davis Drive
Media, Pa. 19063

Invoice:

SOLD TO

Lehigh University
Eaulsion Polymers Institute
Francis Mac Donald Sinclair
Memorial Laboratory #7
Bethlehem, Pa. 18015

INVOICE: 063087
DATE: 30 JUNE 1987

DATE OF ORDER	CUSTOMER PURCHASE ORDER	DESCRIPTION	UNIT PRICE	TOTAL
12/18/86	025379			
		Invoice date: 06/30/1987 Covers effort under PO above for period from 05/31/87 to invoice date.		
		Description of effort: 1. Consultation		
		Design Consultation None		
	2. Design	Mechanical Design None		
		Electrical Design None		
	3. Fab Review	Review @ LU 3.5 hours	$50/hr	$175.
	4 Program Management	Progress report(task 3) 2 hours	$50/hr	$100
		T&L UF to LU June 1 trip 106 mi @ $0.21/mi +$2.20 tolls/trip		$24.46
		TOTAL		$299.46

I certify that the above hours are correct to the best of my knowledge.

Francis A. Vicente
President

APPENDIX C

M S C INC.

2171 DAVIS DRIVE
MEDIA, PA 19063

31 July 1987

Lehigh University
Emulsion Polymers Institute
Francis MacDonald Sinclair
Memorial Laboratory #7
Bethlehem, Pa 18015

Attn. Dr. J.W. Vanderhoff
Director

AUG 04 1987

Dear Dr. Vanderhoff,

This submission constitutes our eighth monthly progress report under Lehigh University's P.O. 025379, dated 18 December 1986. It covers the period from 30 June 1987 through 31 July 1987.

Technical
Support to LU was continued in regard to equipment/ parts acquisition. Clarification and interpretation of drawings and parts lists was given to permit and facilitate ordering of parts to the drawings. In addition support was given to an inspection of approximately 75% of the mechanical parts on 11 July. A followup inspection of other major parts was held on 29 July.
It was found that all parts had been satisfactorily constructed except for the cylindrical insulation cover. This part was not constructed to the released drawing. A too thick (1/4 in.) cylinder was used and not machined down as called for in the drawing. This makes the part heavy. It does not currently affect the performance except for handling and possibly requiring a heavier duty drive motor. Consequently we recommended holding off purchasing the drive motor until assembly is complete and the impact assessed.
The second difficulty encountered was with the piston. The piston shaft did not fit into the LVDT housing assembly. We recommend that both the housing and the piston be sent to the housing vendor with instructions to:

1. measure both the housing cylinder and piston shaft
2. machine the housing cylinder to provide a slip fit between the two
3. repeat for all three sets matching components if necessary and tagging as appropriate
4. measure the new housing cylinder I.D. and report it to LU and reallodine the machined part.

Based on technical discussions with Dr. David Sudol on July 11, the use of teflon coating on the cylinder came into question. Evidently he has encountered some contamination in recent experiments caused by teflon for some of the reactions you are planning to undertake. We recommend that a possible avoidance of this be to polish the inside of the cylinder to a very high finish, namely 16 or better (8 being better yet). This should improve the sliding of the "o" ring, although not as much as using teflon. If the polishing to a high finish does not produce the desired result teflon can always be added later.

Programmatic
A meeting was held at Lehigh U. on 11 July 1987 to discuss construction, parts acquisition and inspect parts. Some parts appeared not to be constructed per our drawings. A recommendation as to course of action is discussed above in the Technical section.

An invoice for the effort to date is attached.

We continue to hold beginning construction of the electrical s/s until all the required electronic parts are available.
If you have any questions please feel free to call (215) 565-2947.

Sincerely,

F.A. Vicente
President

M S C Inc

2171 Da- is Drive
Media, Pa 19063

Invoice:

SOLD TO

Lehigh University
Emulsion Polymers Institute
Francis Mac Donald Sinclair
Memorial Laboratory #7
Bethlehem, Pa. 18015

INVOICE: 073187
DATE: 31 JULY 1987

DATE OF ORDER	CUSTOMER PURCHASE ORDER	DESCRIPTION	UNIT PRICE	TOTAL
12/18/86	025379			

Invoice date: 07/30/1987 Covers effort under PO above for period from 06/30/87 to invoice date.

Description of effort: 1. Consultation	Design Consultation None		
2. Design	Mechanical Design None		
	Electrical Design None		
3. Fab Review	Review @ LU 10 hours 6 hours	$50/hr $33/hr	$500. $198.
4.Program Management	Progress report(task 3) 2 hours	$50/hr	$100
	T&L VF to LU July 2 trip 106 mi @ $0.21/mi +$2.20 tolls/trip		$48.92
	TOTAL		$846.92

I certify that the above hours are correct to the best of ~~my~~ knowledge.

Francis A. Vicente
President